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The Standard Calibration Instrument Automation System for the Atomic Absorption Spectrophotometer Part I: Functional Specifications

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THE STANDARD CALIBRATION INSTRUMENT AUTOMATION SYSTEM FOR THE ATOMIC ABSORPTION SPECTROPHOTOMETER PART 1 - FUNCTIONAL SPECIFICATIONS

by

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This document contains a project definition, a set of functional requirements, and a functional design for the automation of flameless atomic absorption (AA) spectrophotometers.

The system is a real-time data acquisition system with "on line" data reduction, quality control and report generation. The system will automate single or double beam instruments, with or without background correction, and with or without automated sample changers. The data reduction mode of calculation will be method of standard calibrations.

Sample tracking and laboratory data management are integral parts of the system due to direct communication links between the instrument processor and the SFC processor. Sample backlogs from the SFC processor are accessed by the instrument programs for run initialization. Finished data from the instrument program is approved and transferred to the SFC data base.

Two related publications are "The Standard Calibration Instrument Automation System for the Atomic Absorption Spectrophotometer Part II - User's Guide" and "The Standard Calibration Instrument Automation System for the Atomic Absoprtion Spectrophotometer Part III - Program Documentation."

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FOREWORD

Environmental measurements are required to determine the quality of ambient waters and the character of waste effluents. The Environmental Monitoring and Support Laboratory - Cincinnati, conducts research to:

- Develop and evaluate techniques to measure the presence and concentration of physical, chemical, and radiological pollutants in water, wastewater, bottom sediments, and solid waste.
- Investigate methods for the concentration, recovery, and identification of viruses, bacteria and other microbiological organisms in water; and to determine the responses of aquatic organisms to water quality.
- Develop and operate an Agency-wide quality assurance program to assure standardization and quality control of systems for monitoring water and wastewater.
- Develop and operate a computerized system for instrument automation leading to improved data collection, analysis, and quality control.

This report was developed in the Advanced Instrumentation Section of the Environmental Monitoring and Support Laboratory (EMSL) in the interest of advancing laboratory techniques and quality control through computerization.

Robert L. Booth
Acting Director
Environmental Monitoring and
Support Laboratory - Cincinnati

ABSTRACT

This document contains a project definition, a set of functional requirements, and a functional design for the automation of atomic absorption (AA) spectrophotometers.[1] The EPA laboratory automation system[2] host processor is the Data General Nova series mini computer and the Sample File Control (SFC) host processor is the Digital Equipment Corporation PDP 11/70 mini computer.

The system is a real-time data acquisition system with "on line" data reduction, quality control and report generation. The system will automate single or double beam instruments, with or without background correction, and with or without automated sample changers. The data reduction mode of calculation will be method of standard calibrations.

Sample tracking and laboratory data management are integral parts of the system due to direct communication links between the instrument processor and the SFC processor. Sample backlogs from the SFC processor are accessed by the instrument programs for run initialization. And finished data from the instrument program is approved and transferred to the SFC data base.

The system provides full automation capabilities for an USEPA laboratory interfaced into the EPA Data General/Digital Equipment computer automation scheme.

Two related publications are "The Standard Calibration Instrument Automation System for the Atomic Absorption Spectrophotometer, Part II - User's Guide" and "The Standard Calibration Instrument Automation System for the Atomic Absorption Spectrophotometer, Part III - Program Documentation." This report covers a period from November 1978 to February 1980 and work was completed as of February 1980.

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SECTION 1

INTRODUCTION

These functional specifications describe an automation package for the flameless atomic absorption spectrophotometers. The report is divided into four main areas: project definition, overview of the project, requirements of the system, and functional design. The combination of these four areas describe the system to a sufficient degree so as to allow an operator to decide if the specified system will meet his requirements.

The project definition is the agreement between users, implementors, and management as to the scope of the project. It contains a brief written statement of the overall goals of the project.

The overview of the project contain a brief description of flameless AA spectroscopy, a description of the existing system and instrumentation involved and any technical constraints imposed on the proposed system. The support issue is also addressed under the nontechnical constraints to allow management to be aware of the additional person power requirements or reliance on resources outside the laboratory in which the instrument was automated.

The requirements of the system contain a detailed list of the requirements of the automation system. This includes hardware requirements, calculation requirements, reporting requirements, and all interaction necessary with the system to obtain the desired objectives in the project definition. Enough information must be contained in the functional requirements to allow a functional design to be formulated. The functional design is a "blueprint" of the system showing a flowchart of the order of operations, and a detailed description of all input, output, and human interaction. The functional requirements are used to produce a design framework linking all the functional blocks. The functional design is then used in the implementation phase which entails producing the detailed design, building the appropriate hardware and software modules, and testing the system components.

The final system will provide full documentation of all hardware and software modules and a users manual for operating the automation system.

These functional requirements were written to satisfy the needs of Region III, Region V, Municipal Environmental Research Laboratory, Cincinnati (MERL), and Environmental Monitoring and Support Laboratory, Cincinnati (EMSL). An overall summary of the benefits of the AA automation package are:

- to increase the number of samples which may be measured by the analyst
- to increase the accuracy and precision of the data reported
- to facilitate the movement of sample file control data to and from the SFC computer.

SECTION 2

PROJECT DEFINITION

The goal of this project is to develop a computer automation system which will accept raw data from a variety of commercially available flameless atomic absorption spectrophotometers and process the data. The system will interact with the EPA sample file control system so as to allow bidirectional communication between the instrument program and the sample file control system. The system will provide:

- real-time data acquisition from the AA units
- "on line" standard calibration and concentration computation by interpolation, linear regression, second degree regression, third degree regression, or a forced-zero linear regression,
- "on line" quality control during the course of the run to signify, discrepancies to the analyst as they occur,
- system adaptability to double or single beam instruments, operating with or without background correction,
- interaction with the sample file control system for transferring samples from a backlog file to the instrument run file (sample wheel pattern), and transferring final results back to the sample file control system
- unattended data acquisition from AA automatic sample changers
- the capability to stop automatic sequencing during the run and revert to an operator controlled system,
- The capability to start or restart an automatic sequencing procedure at a user defined wheel position,
- manual sequencing procedures for instruments without automatic sample changes,
- replicate statistics on multiple measurements of any sample,
- dilution and remeasurement options,
- editing of results prior to final reporting,
- detailed final reports on samples and quality control,
- plotting of calibration standards and raw data.

SECTION 3

OVERVIEW OF THE PROJECT

3.1 NATURE OF THE FLAMELESS AA LABORATORY EXPERIMENT

3.1.1 Scientific Principles of Flameless Atomic Absorption Spectroscopy

The flameless atomic absorption instrument, sometimes referred to as a graphite furnace or carbon rod analyzer, is used to determine low level concentrations of metals in environmental samples.

The atomic absorption spectrophotometer consists of the following components: a characteristic atomic-line source (hollow-cathode lamp) of the metal to be determined, a high temperature furnace (flameless source) to produce metal atom vapor from the sample, a monochromator to select light of the appropriate wavelength, and a photomultiplier tube to detect the light. (See reference 3 for the automated flame AA functional requirements.) Figure 1 illustrates the major components of most flameless AA instruments.

The basic steps in a single determination requires a sample injection into the furnace chamber, a low temperature heat step to evaporate the solvent, a high temperature heat step to break down the residue, and a very high temperature heat step for the atomization of the sample. At the point of atomization, the detector records a decrease of transmitted light through the sample, or an increase in absorbance of the light caused by the neutral atoms of the metal of interest absorbing the characteristic light from the hollow cathode source. Beer's law holds true in this type of experiment since the absorption measurement of an atomized sample taken at the appropriate wavelength is generally proportional to the concentration of the particular metal in the sample.

3.1.2 Description of the Existing System

The analyst conducting the experiment utilizes methods manuals,[4] experience, and manufacturer's guidelines for preparing samples and optimizing the instrument and associated equipment. It is assumed that the chemical methods are well defined for each analyte, and each type of sample being measured. Instrument set up and optimization, however, may be generally described irrespective of the particular type of instrument being used. The main features of the set up procedure involve a number of tasks. These include the adjustment of light source voltage level(s), the measurement of distances between source, sample, detector and intervening lenses, wavelength and slit width settings, temperature and time settings for the reduction of the sample, auto sampler setup, inert

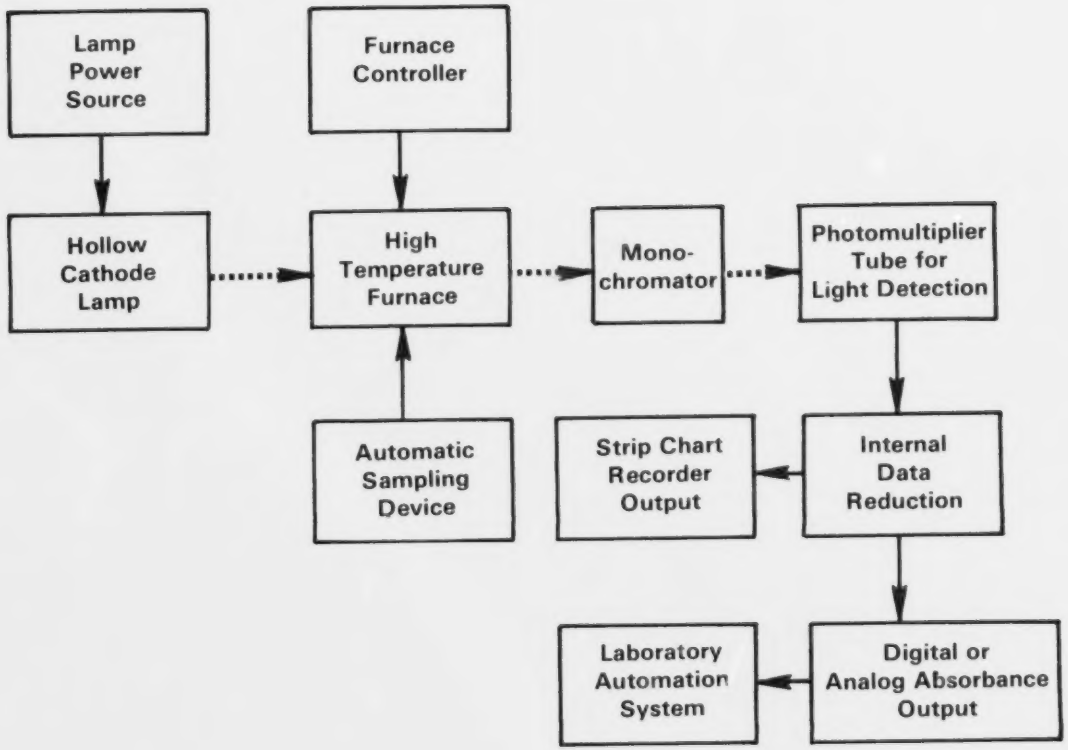


Figure 1. Main components of the flameless atomic absorption instrument.

gas settings, and associated adjustments of output devices such as digital recorders and strip chart recorders. These procedures are defined for each instrument and analyte.

After the initial calibration and setup of the instrument, the analyst procedes to the actual analysis of the samples. In general, there are two computational methods for determining metal concentrations in environmental samples using the flameless AA. The methods are the standard calibration approach and the standard additions approach. Most samples that produce poor spike recoveries using the standard calibration method may be evaluated using the standard additions method. This is a more time consuming approach and therefore less desirable, however, the method is mandatory for samples with matrix interferences. This report covers only the standard calibration method. The method of Standard Additions will be discussed in detail in future documents.

3.1.3 Unautomated Method of Standard Calibration

The Standard Calibration mode of analysis utilizing the flameless AA requires the measurement of a number of standard solutions and the development of a standards curve relating absorbance signals to known or standard concentrations. In the non-automated experiments the curve is obtained by measuring the height of each standard atomization peak on the strip chart recorder or obtaining a corresponding reading from a digital panel meter, and plotting these absorbance measurements against the known concentrations of each standard. Once the analyst is satisfied with the calibration curve, measurement of the unknown samples begins.

An unknown sample atomization peak is measured, and the absorbance is used to calculate the concentration of the particular analyte in the sample. If a sample atomization peak is beyond the limits of the calibration curve, a dilution is made and the analysis continues until the sample peak is within the calibration region.

3.1.4 Quality Control Measurements

Quality control measurements must be made during the course of the run in order to verify instrument stability (instrument check standards), method precision (duplicate unknowns), and sample matrix non-interference (spiked unknowns).

At various stages in the run, the analyst may choose to measure a standard in order to verify the integrity of the original curve within the time frame of the analysis. This type of measurement is referred to as an instrument check standard, and it is a quality control test on instrument stability. A similiar type of check standard is supplied by the laboratory and undergoes all sample processing. This is a laboratory control standard, and it is a quality control test on method stability and the quality of the standards.

Duplicate unknown measurements may be made on two independently processed aliquots of a sample. Normally, significant differences in

these two measurements indicate errors in the method or turbidity problems.

Laboratory reagent blanks are measured to determine if there is an absorbance due to the sample preparation technique. The reagent blank concentration may be subtracted from all samples prepared by the specific technique.

Another type of quality control sample is the spiked unknown. Variations in the expected spike recovery normally indicate problems related to matrix interference in the samples themselves or to accuracy problems with standards.

In some cases, replicate measurements are made on the samples and average signals and replicate statistics are calculated. Outliers may then be eliminated and more precise calculations may be made.

3.2 Instruments to be Automated

The following Perkin Elmer atomic absorption spectrophotometers can be operated with or without background correction and are to be automated:

- Perkin Elmer Model 303
- Perkin Elmer Model 503
- Perkin Elmer Model 403
- Perkin Elmer Model 5000

The above instruments can be operated with or without a Perkin Elmer Model AS-1 sample changer. The Perkin Elmer Model HGA 2100, Model HGA 2000, and HGA 500 furnace controllers are used to control the high temperature furnace on the above instruments.

The following Varian instruments which can also be operated with or without background correction are to be automated:

- Varian Model AA-5, AA spectrophotometer
- Varian Model A-6, AA Spectrophotometer with a Model ASD-53 Automatic Sample Changer

The above instruments use the Varian model 63 carbon rod atomizer for sample atomization.

3.3 NONTECHNICAL CONSIDERATIONS AND CONSTRAINTS

3.3.1 Time and Financial Considerations

The first version of the Standard Calibration AA automation system with SFC interface should be operational by January 1, 1980. Complete documentation of the automation package should be completed in a final report form by March 15, 1980.

Total effort should take less than 0.5 person year. Therefore, the cost for functional specifications, implementation design and final documentation should not exceed $25,000. This figure includes $15,000 of programming effort of outside contractor and $10,000 of "in house" effort reflected in system analysis and system engineering.

The initial software and hardware development and implementation cost can be distributed over additional instruments which are interfaced to the AA system. The software can be transferred at practically no cost and the previously designed hardware can be transferred at minimal individual instrument cost. Individual hardware cost for interfacing a particular instrument should not exceed $1000 excluding the communication terminal. This includes modifying the instrument for raw data acquisition, and installing any additional custom hardware for digital input or control. Travel and training are not included in the above cost.

3.3.2 Support of the Completed System

Support of an automated system is divided into software and hardware support. In each of these areas the manufacturers equipment and software were left "intact" as much as possible. This allows independence from any custom hardware or software packages. The complexity of the AA automation system, however, does require the need for some custom hardware and software. Therefore, this section describes the responsibilities for the maintenance of the various sections of the total system.

The Data General NOVA computer system excluding the digital interface and the analog interface is the responsibility of the Data General Corporation through a maintenance contract which must be maintained by the laboratory utilizing the system. All custom hardware must be removable to allow the Data General service engineers to have a clean system to troubleshoot. Data General maintains the peripherals, central processing unit, memory, etc.

The Data General RDOS 6.x and Basic Rev. 4.3 software are the responsibility of Data General. Modifications to the Basic Rev. 4.3 sources are the responsibility of the USEPA-Cincinnati.

Any custom hardware on the AA automation system is the responsibility of the Advanced Instrumentation Section, EMSL-CI for a period of one year after delivery. Mandatory interfacing procedures for instruments require a full set of documentation with any custom interface hardware in addition to a complete set of spare cards. This allows "in house" repair of custom hardware through module swapping.

The Basic application code provided by the USEPA-Cincinnati for the automation of various instruments is supported by the USEPA-Cincinnati for a period of one year after delivery. Programming procedures require a complete set of documentation on the application code in addition to a users manual. It must be stressed at this time that USEPA-Cincinnati

will provide a standard software application package to requesting laboratories, however, changes in the standard package prohibit the maintenance of software modified external to USEPA-Cincinnati. Training on the use of the automation system will be provided by USEPA-Cincinnati.

3.4 SPECIFIC HARDWARE/SOFTWARE IMPLEMENTATION REQUIREMENTS

The flameless AA project is one branch of a much larger laboratory automation project. Therefore, some of the basic decisions about system hardware and software have already been made within the context of the larger project. Thus, these functional requirements are not strictly hardware and software independent.

This project will use an existing Data General NOVA 840 minicomputer with the operating system RDOS (Revision 6.2 or 6.x). The RDOS operating system will be upgraded until the current version of the extended Basic system is not supported.

The multiuser extended Basic system is currently frozen at Revision 4.3. This is the result of extensive modifications to the sources. That is, the Data General Basic has been modified by Lawrence Livermore Laboratories to allow real-time data acquisition and control through calls to assembly language routines. Data reduction, operator control, and report generation are accomplished through the Basic high level language.

Due to the cost in modifying the Basic sources and associated software, a move to another version of Data General's Basic will only occur if the present revision fails to satisfy the needs of the agency.

The NOVA 840 minicomputer was interfaced to a general purpose interface card to provide digital input and output (I/O) control. All inputs and outputs are controlled from the Basic language. Any instrumental control will, therefore, be implemented with the existing digital I/O hardware. Likewise, a fourteen bit analog to digital (A/D) converter was interfaced to the system for a multiplexed input of up to 32 channels of analog input. This preceeding design should also be utilized for the acquisition of analog data. This should not discourage the input of digital data. Multiplexers have been inserted into the standard digital interface for future expansion to digital types of inputs.

3.5 FUTURE CONSIDERATIONS FOR THE FLAMELESS AA SYSTEM

The Flameless Atomic Absorption System is being designed to accomodate the majority of commercially available instruments. It is the expectation of the designers that data from these instruments will be available either in the form of a digitized array of peak absorbance data or as a fully reduced single-number absorbance value. Any further reduction by "smart" instruments is considered useless due to the complex interlationship of samples necessitated by Quality Control requirements and Sample File Control (SFC) restrictions.

The Flameless AA system will be easily adaptable to any type of interfacing scheme and as such will be compatible with most Flameless AA instruments. Further explanations regarding this subject may be found in the Raw Date Acquisition Requirements of Section 4.

SECTION 4

REQUIREMENTS OF THE SYSTEM

In this section, the major functions of the atomic absorption instrument automation system will be described. System requirements and constraints will be specified for each functional area. In order to view the functional areas in the perspective of an automated analysis, some terms require definitions.

An analysis sequence or run is defined as an experiment within which an analyst may measure a group of samples composed of standards, unknowns, and quality control items within the context of a particular analytical method. The sequence of sample measurement is predefined before the run begins and may be viewed as a sample wheel pattern even if no automatic sample changer exists.

A fully automatic sequence refers to instrument systems which utilize an automatic sampling device. When such a device is available, it will be possible for the analyst to initiate an automatic run which will: 1) cause the measurement of standard samples, 2) perform calibrations, 3) measure unknown samples, 4) display interim results as they are calculated, 5) measure QC samples, and 6) evaluate QC acceptability. Since automatic sampling devices generally allow the option of setting a replicate injection counter before the run, the automated instrument system will allow for a set number of replicate injections (between 1 and 6) of each sample during the fully automatic analysis. Once the analysis has been completed, the system will revert to the semiautomatic sequencing mode in order to allow for additional sample measurement, replicate deletion, changes in dilution factors, recalibration, and updated reporting of run results. This semiautomatic sequencing mode will still utilize the sample changer to control injections but it will require some manual intervention on the part of the analyst to: 1) position the wheel if further sample measurements are required or 2) input commands to perform non-measurement functions.

The analyst who has the use of the automatic sample changer may elect to perform the entire analysis in the semiautomatic sequencing mode. The analyst will be required to identify the position of the sample being measured before the measurement takes place. Replicate absorbance measurements of the same sample will still be allowed under operator control. When non-measurement functions are desired, such as replicate deletion, changes in dilution factors, re-calibration or reporting, the analyst must type a command indicating the function he wishes performed.

In cases where no automatic sample changer is available, the analyst will still be required to measure samples using the sample wheel pattern which identifies the location and type of samples in the analytical sequence. This type of run will be called an operator assisted semiautomatic run since the analyst will be required to manually inject the sample, and activate an instrument control which will start the atomization procedure for the sample measurement. The analyst will still be allowed to perform non-measurement functions in this mode using the appropriate commands.

For the sake of continuity, only the automatic and semiautomatic sequencing procedures will be referred to in the following discussion.

An additional clarification must be made regarding the term "automated instrument system." Whether or not the instrument has an automatic sample changer, the instrument is still "automated" since atomic absorption data is acquired "on-line" and without any operator intervention.

4.1 RAW DATA ACQUISITION REQUIREMENTS

It is required that real-time data acquisition hardware (and corresponding system software) be made available for three unique types of Flameless AA output. These include high speed data sampling for both double and single beam instruments such as the Perkin-Elmer 503 and Varian AA-5. The system is also required to receive fully reduced absorbance data through instruments commercially equipped with EIA-RS-232C interface capabilities.

Since the newer atomic absorption instruments have highly sophisticated electrical and computational data acquisition and reduction systems, it has been decided to utilize these capabilities and thus relieve the data reduction burden currently placed on the lab automation computer. Hardware and system software is currently designed and is operational at EMSL-CI[5] to perform the task of acquiring this type of output.

The requirements for data acquisition and reduction using older single or double beam instruments are stated in the following paragraphs.

Data should be acquired at a rate sufficient to allow the analysis of the fastest peak being produced by the flameless AA technology. The sampling rate must be sufficient to define the leading and trailing baselines as well as the shape and height of the peak itself. Normally a minimum of fifty points are required to define a peak.

The system must be synchronized to the instrument to avoid ever missing any data peaks generated by the instrument. The synchronization should require little or no operator intervention during the course of an analysis sequence.

When the automatic sample changer is used, the hardware of the automatic sampler in some cases must be controlled by the computer for proper operation of the AA system. Without computer control of the automatic

sampling device, atomization could occur before the computer data buffer is available for incoming data. This would result in the loss of the data from the instrument. To insure data integrity the automatic sampling device must be prevented from injecting the next sample until the computer data buffer is available for incoming data unless another type of buffering device is utilized.

The operator must have the capability to disable computer controls on the automatic sampling system, therefore all custom hardware should be designed in such a fashion as to allow removal of computer controls in order to return the instrument to a vendor supported state.

The current concensus regarding the interfacing of the newer AA instruments which produce a fully reduced absorbance value is that the lab automation computer must not impede the performance of the instrument. In other words, a PE-5000 should run unimpeded through an entire sample wheel without data loss.

4.2 RAW DATA REDUCTION REQUIREMENTS

Raw data acquired during a sample atomization cycle must be converted to a single absorbance value. This value will be used in subsequent calculations in order to calibrate standards or compute sample concentrations. The raw instrument data may be in the form of a digitized transmittance signal or a digitized absorbance signal acquired over the duration of the atomization cycle.

The system is required to convert each point in the transmittance array to an absorbance value through the equation:

$$\text{Absorbance} = \text{Log}_{10} \frac{\text{Treference}}{\text{Tsample}}$$

If the AA instrument is double beam, the reference signal will represent either a split source beam shunted around the sample or a hydrogen (deuterium) correction beam shown through the sample. If the AA instrument is single beam, no reference signal will be measured and the T reference will be a constant.

The system is required to output this absorbance array in the form of a plot to a lineprinter or the user terminal for pre-run timing verification. It is also required that every absorbance array, collected during the analysis, be stored for plotting on a graphics display terminal.

The system is required to evaluate the peak height of the absorbance signal. This will be done by performing a quadratic least-squares fit on the five top most points in the array, computing the maximum point from the first derivative of this equation, and subtracting the signal baseline from the maximum value. This value will constitute the absorbance signal of the sample and must be displayed "on line" at the user terminal. On an unloaded system the reduction of peak data into a single absorbance value must be accomplished within five seconds after the atomization has been completed.

Further data reduction to a concentration value and/or a quality control check must be accomplished within five seconds after the absorbance value has been obtained.

4.3 STANDARD CALIBRATION REQUIREMENTS

In order to compute the concentration of an analyte in environmental samples using the standard calibration method, a set of standards with known concentrations must be analyzed first. Each standard will exhibit an absorbance value. The set of known standard values and their associated absorbance measurements may be used to produce an equation relating concentration to absorbance.

At least one standard must be measured in any run before a calibration equation can be generated. Up to twenty different standards may be measured and calibrated. As many as six replicate measurements may be made on any standard, and all of the standard measurements will be used in the standard calibration algorithm.

A number of computational modes will be available to the analyst prior to making the run. The calibration standards may be fit to an equation using one of five techniques: 1) first degree least-squares regression; 2) first degree least-squares regression forced through the origin; 3) second degree least-squares regression; 4) cubic least-squares regression; or 5) linear interpolation between two bracketting standards.

The system will be required to automatically calibrate the standards in a fully automatic analysis sequence and be able to calibrate (at the user's request) in the semi-automatic analysis sequence.

When calibration is performed, the system will be required to output a calibration report to an operator selected output device. This output will include the computed calibration equations and the fitting errors for each of the calculated equations. It will also include a table showing the relative absorbance values, the known standard values, the computed concentrations for each standard (from the calibration equation), and the difference between the known and computed concentrations for each standard. The system will also be required to save the calibration information "on line" in order to facilitate the plotting of the points and the equations on a graphics display terminal.

Since only one computational mode can be utilized at any given time to evaluate sample concentrations, the system is required to obtain a preliminary computational mode from the analyst before the run begins. The selected mode will be used throughout an automatic sequence to compute sample concentrations. The system is required to produce the calibration report as soon as calibration occurs (normally after the last calibration standard is measured). Depending on the type of graphic support devices available on the system, this report and corresponding plots may be scrutinized during the remainder of the run. As soon as the automatic run is complete, the system is required to facilitate the easy transfer from the pre-selected computational mode to a more appropriate mode.

4.4 QUALITY CONTROL REQUIREMENTS

Two major quality control (QC) categories are applicable in most analytical experiments. Laboratory QC is used to monitor accuracy, precision, and detect erroneous measurements caused by many possible conditions including matrix interference or turbidity effects. Laboratory QC samples are always treated exactly the same as unknown samples in that they receive the same pre analysis chemical processing. Instrument QC is used by the analyst in monitoring instrument stability throughout the analytical sequence. The following QC types have been developed to fulfill the objectives for laboratory quality control (see Table 1):

- lab reagent blank (LRB)
- lab control standard (LCM, LCT)
- lab spiked sample (LSO, LSF, LSA)
- lab duplicate sample (LD1, LD2)

These sample types have been defined in the Sample File Control Data Element Dictionary.[6] Some additional techniques have been defined for the maintenance of precision and instrument stability and are identified as follows:

- calibration check standard (CS)
- replicate measurements and statistics
- off-scale sample notification

4.4.1 Laboratory Quality Control

One of the major objectives of instrument automation is the "on line" evaluation of acceptability for any quality control sample being measured during a run. In order to fulfill this objective, three functions will be required of the automation system. First, the system must allow for the pre run definition or selection of limits by which to evaluate lab control standards, lab spikes, lab duplicates, and lab reagent blanks. Second, the system must be capable of computing a QC value "on line" for any of the lab QC items being measured during the run. Third, the system must be able to evaluate QC acceptability by comparing the QC value with the defined limits and report whether the result is an in control, a warning, or an out of control measurement.

The acceptability of quality control results computed for laboratory QC samples will be evaluated relative to limits defined by the analyst or historically maintained by the system through Sample File Control.

The limits for the four major QC sample types are described as follows:

- for lab reagent blanks
  the mean recovery ($\bar{X}$)
  the lower and upper warning limits (LWL, UWL)
  the lower and upper control limits (LCL, UCL)

- for lab control standards
  the mean percent recovery ($\bar{X}$)
  the lower and upper warning limits in % (LWL, UWL)
  the lower and upper control limits in % (LCL, UCL)

- for lab spikes
  the mean percent recovery (X)
  the lower and upper warning limits in % (LWL, UWL)
  the lower and upper control limits in % (LCL, UCL)

- for lab duplicates
  the slope (a) and intercept (b) terms for the equation:

$$R_c = a\bar{X} + b$$

The computation of QC values will be based on the type of QC sample being measured. Each QC value will be compared with the associated QC limits in order to evaluate QC acceptability. The following QC calculations and assessments will be made and displayed to the user terminal:

- Laboratory reagent blanks will require computation and display of the calculated sample concentration (LRB); this value will be compared with the reagent blank limits and the appropriate QC judgement will be displayed to the user terminal.

- Laboratory control standards will require computation and display of the measured concentration (LCM), the theoretical concentration (LCT), and the percent recovery of the sample, where:

$$\%\ \text{Recovery of lab control standard} = \frac{LCM}{LCT} \times 100\%;$$

  this value will be compared with the lab control standard limits and the appropriate QC judgment will be displayed to the user terminal.

- Laboratory spikes will require the computation and display of the original sample concentration (LSO), the spiked sample concentration (LSF), and the percent recovery of the spike (LSA), where:

$$\%\ \text{Recovery of lab spike} = \frac{LSF-LSO}{LSA} \times 100\%;$$

  this value will be compared with the lab spike limits and the appropriate QC judgment will be displayed to the user terminal;

- Laboratory duplicates will require the computation and display of the original unknown concentration (LD1), the concentration of its duplicate (LD2), the difference between the two concentrations (R), and the average of the two concentrations (X), where:

$R = LD1-LD2$ and $\bar{X} = \frac{LD1+LD2}{2}$;

$\bar{X}$ will be substituted into the previously defined critical range equation to produce R_c; an out of control judgment will be displayed to the user terminal if R is greater the R_c.

4.4.2 Instrument Quality Control

The system will be required to monitor Instrument QC throughout the computer assisted analysis. Instrument check standards will be measured to validate instrument stability. A standard of this type will be identical to one of the calibration standards, and will be evaluated in the following manner:

$$\% \text{ Recovery of Check Standard} = \frac{\text{check standard abs.}}{\text{calibration std.abs.}} \times 100\%.$$

This percent recovery will be used to notify the analyst of any drastic changes in the instrument conditions. The analyst will be required to define the control limits for this evaluation prior to the analysis. If the limit is exceeded during the analysis, the system will notify the analyst. If a fully automatic run is in progress when the check standard limit is exceeded, automatic sequencing may be stopped by the analyst and the system will revert to the semi-automatic mode and wait for a user command. The analyst may then generate a status report and abort or continue the run in the semi-automatic or fully automatic mode.

The system will facilitate the repeated measurement of any sample in the analytical sequence to appraise the analyst of instrument precision. Each time a sample is measured, the system will display the measured absorbance, the average relative absorbance of the sample, the number of replicates maintained for the sample, and each replicate absorbance value. In subsequent analysis reports, the average concentration, and the standard deviation will be presented on any sample which was measured more than once.

The system will also be required to notify the analyst any time a sample absorbance falls below the absorbance of the lowest non-zero calibration standard or above the absorbance of the highest calibration standard.

The system is required to facilitate dilution and remeasurement procedures for off-scale samples.

4.5 COMPENSATION FOR DILUTION

Environmental samples may be diluted to reduce their concentrations to a level within the range of an analytical method. The measured concentration of such a sample must be adjusted so that the reported concentration is that of the original sample rather than the diluted sample. For the automation system to make this adjustment, the analyst will have to enter the volume of the original sample and the volume of the dilution water. The system will calculate the concentration of the original sample by the formula.

$$C_o = C_m \times \frac{V_o + V_d}{V_o}$$

Where C_o is the calculated concentration of the original sample, C_m is the measured concentration of the diluted sample, V_o is the volume of the original sample, and V_d is the volume of the dilution water.

Dilution procedures are complicated somewhat when diluted samples are involved in quality control sets. Obviously, in the case of duplicate samples, both members of the duplicate pair must be diluted identically, and the system will use the formula above to calculate the values reported as LD1 and LD2.

The formula above will also be used to calculate the values for LSO and LSF in cases of diluted samples which are spiked with an insignificant volume of spiking solution. The system will require that dilution be done before spiking. The value for LSA will be calculated by the formula.

$$LSA = \frac{M}{V_o}$$

Where M is the mass of the analyte added in the spike and V_o is the volume of the original, undiluted sample.

For analysts who prefer to use spikes with significant volumes, the following formulas must be used to calculate the relevent values for spikes of diluted samples.

$$LSO = C_m \times \frac{V_o + V_d}{V_o}$$

Where C_m is the measured concentration of the diluted but unspiked aliquot, V_o is the volume of that aliquot before dilution and V_d is the volume of the dilution water.

$$LSF = C_m \times \frac{V_o + V_d + V_s}{V_o + V_s}$$

Where C_m is the measured concentration of the diluted, spiked aliquot, V_o is the volume of that aliquot before dilution or spiking, V_d is the volume of the dilution water, and V_s is the volume of the added spiking solution.

$$LSA = \frac{C_s V_s V_o - C_m V_o V_s - C_m V_s V_d}{V_o V_o + V_o V_s}$$

Where C_s is the concentration of the spiking solution, C_m is the measured concentration of the diluted but unspiked aliquot, V_s is the volume of the spiking solution, V_o is the volume of the undiluted, unspiked aliquot, and V_d is the volume of the dilution water.

A detailed justification for these formulas can be found in the memorandum "Proposed Sample Dilution Procedures and Sample Spiking Procedures for Sample File Control" by Dennis Ryan, Jonathan Kopke, Gerald McKee, and Dr. William Budde.

4.6 MAJOR RUN-TIME CONTROL FUNCTIONS

The analyst will have the responsibility for setting up and directing the course of the computer assisted analytical sequence. He will be required to define the sequence and type of samples to be measured. He will also be responsible for defining specific timing parameters, the instrument type, and the method of computation used during the analysis. Once a run has been defined, and the sample wheel pattern has been obtained, the analyst may measure each sample through the semiautomatic sequencing procedure. In cases where an automatic sampling device is available, the analyst may initiate a fully automatic sequencing procedure which will acquire, reduce, and display results on all of the samples in the pre-defined sample wheel pattern.

The system will be required to perform a number of control functions during the course of the run. These functions may be initiated through the operator controlled semi-automatic mode or may be generated automatically during a fully automated run. Briefly, the required functions of the system are as follows: sample measurement, calibration of standards, interim report generation, sample editing, and final report generation. Each of these major functions, along with some miscellaneous functions, will be described in more detail in sections 4.6.1 through 4.6.6.

4.6.1 The Measure Function

The sample measurement function will facilitate the acquisition of an absorbance value, reduce the absorbance value to a concentration through a predetermined calculation mode, and output pertinent parameters for a sample in the wheel pattern. The sample will be identified by its wheel location or sequence number. The wheel pattern will contain all of the information needed to characterize the type of sample being measured. This information will be used by the system to define the form and content of the interim report required for each type of sample measurement. Table 1 shows the various types of samples which may be measured in a run and defines the type of output which may be expected "on line" after a measurement of each sample type.

4.6.2 The Calibration Function

The calibration function will facilitate the development of a number of equations relating concentration to absorbance using the least squares regression technique. In general, the analyst will define the mode by which concentrations will be computed before the analysis begins. After the calibration standards have been measured, the equations will be developed and the appropriate equation will be used to compute subsequent sample concentrations. A plot of the standard points and the calibration equation will be available to the analyst any time after calibration has been

performed. At any time during the semi-automatic mode of operation a recalibration may be made.

4.6.3 The Interim Report Function

The report generation function will facilitate the output of a hard-copy report signifying the status of the run. Any number of reports may be generated during the run but, in general, the analyst will measure all of the samples in the sample wheel pattern before generating the first report. The report will contain the following information:

Report Heading
- run identifier
- title, time, date
- instrument name, number, and location
- analyst's initials
- method, analyte, and method parameter code
- units of concentration
- comment fields containing instrument conditions and sample information

Standards Heading
- number of standards used
- type of calibration equation used
- calibration equation
- fitting error (% relative standard error of estimate)

Standard Sample Data
- standard sequence number (in wheel pattern)
- number of replicates
- average absorbance for standard
- expected concentration (true value)
- calculated concentration
- difference between known and computed concentrations

Unknown sample data
- sample sequence number in wheel
- laboratory sample identifier
- number of replicates
- average absorbance signal
- average concentration
- off-scale sample indicator
- dilution factor
- undiluted sample concentration
- standard deviation (of replicates, if any)

Quality Control Data
- QC sample sequence in wheel
- QC type
- expected concentration (if applicable)
- QC value (percent recovery or critical difference)
- QC determination (in control, warning, or out of control)

Once a report is generated, the analyst may delete poor standard measurements and recalibrate the standards, or he may dilute and remeasure a sample in the semi-automatic mode with assistance from the Edit function described below.

4.6.4 The Edit Function

The Edit function will only be allowed in the semi-automatic mode and will facilitate the preview and edit of data maintained on a particular sample in the sample wheel pattern. By identifying the appropriate wheel location, the analyst may view all of the replicate absorbance values maintained on the sample. It will also display the dilution factor, computed concentration, and QC information when applicable. The analyst may delete a replicate absorbance or update the dilution factor for the sample. If a replicate absorbance is eliminated, the system will update the average absorbance for the sample and display the updated data on the sample. When a dilution factor is changed, as in the case of an off-scale sample, the absorbance values for the sample and its associated Quality Control sample(s) will be purged. The analyst must then follow dilution procedures and remeasure the sample and the associated QC items.

4.6.5 The Final Reporting Function

The system is required to optionally produce two types of final report. One is specific to sample file control applications and relates to the conversion of the SFC Run File to the SFC Run Results File through the inclusion of sample concentrations determined during a run (Section 4.7). The other type of final report will be produced for non-SFC applications. Either option should be available irrespective of the SFC aspects of the analysis. Both report types will contain sample identifiers, sample concentrations, and method and/or quality control qualifiers. (See reference 6 for a detailed list of result qualifiers.)

The final report function will be augmented by a reagent level subtraction mechanism so that sample concentrations may be adjusted in those cases where there is a uniform method contamination detected in the run.

The final report function will normally be the last activity performed in an analysis run.

4.6.6 Other Miscellaneous Command Functions

Besides servicing the five major command functions mentioned above, the system will be required to respond to other control related commands. The HELP function will be available to the analyst who desires a list of command options. This list will include all of the available run time commands. The system will also facilitate the change of computational modes; it will allow for a restart of automatic sequencing; and it will allow the output mode to be changed.

Plotting will be made available to allow review of the standards curve or raw data peak plots (when applicable). An instrument calibration/ checkout function should be available to allow the analyst the opportunity to verify the integrity of his instrument.

4.7 SAMPLE FILE CONTROL INTERFACE REQUIREMENTS

The Flameless AA instrument automation system will be one of many instrument automation systems which will utilize the bidirectional communication link between the SFC computer system and the automation computer system. The automation computer will communicate with the Master Sample File Control Data Base Management Computer (SFCDBM) to obtain sample backlogs and report sample results.

The major functions of the SFC Interface Computer System will be discussed here, along with specific requirements of the Flameless AA instrument automation system as it relates to the overall structure of Sample File Control.

The main functions of the SFC Interface Computer System are briefly stated as follows:

1) receive and print backlog files and quality control limits obtained from the Master SFCDBM Computer;

2) allow for the selection of sample identifiers from a backlog list for SFC related analysis, or allow for manual entry of sample identifiers for non SFC related analysis;

3) allow for the formation of an analysis sequence (Run File) containing standards, unknowns, and quality control samples through the use of predefined templates;

4) assist in the definition or selection of quality control limits which will be used during the course of a run;

5) facilitate the formation of templates which describe regularly used standard patterns and unknown/QC patterns;

6) when possible, perform an automated analysis of standard, unknown, and QC samples defined in the run sequence (Run File) using an available instrument automation system;

7) perform the automatic recording of results obtained from a fully automated analysis of SFC related samples in a format understandable to the Master SFCDBM Computer, (Run Results File format);

8) assist in the manual or keyboard entry of results obtained from a non-automated analysis of SFC related samples in a format understandable to the Master SFCDBM Computer;

9) allow for the editing and final approval of the results obtained from an SFC related analysis;

10) re-que samples to backlog if unacceptable results were obtained;

11) automatically send the approved run results to the Master SFCDBM computer.

The atomic absorption instrument automation system will be required to fulfill functions 6 and 7 mentioned above. As such, the system will be required to access the defined analysis sequence (Run File) and translate this run pattern into a form compatible with the instrument software. The system will then be required to assist the analyst in measuring the samples as described in previous sections of this document. Once the analyst has generated an instrument report containing satisfactory results, the system is required to automatically record all SFC related results in the form of a run history (Run Results File). The system is required to format this report in a manner compatible with SFCDBM specifications. (6-8)

TABLE 1. MEASURABLE SAMPLES AND RELATED OUTPUT

Sample Type	Sample ID	Replic. ABS	Comp. Conc.	QC Assess.	True Value	Dilution Factor	Off-Scl Deter.
Instrument Blank (B)	NO	NO	NO	NO	NO	NO	NO
Instrument Calibration Standard (S)	NO	YES	NO	NO	YES	NO	NO
Instrument Check Standard (CS)	NO	YES	NO	YES	NO	NO	NO
Unknown Smpl. (bbb, LSO LD1, LDX)	YES	YES	YES	NO	NO	YES	YES
Spiked Unknown (LSF, LSA)	YES	YES	YES	YES	YES	YES	YES
Duplicate Unknown (LD2)	YES	YES	YES	YES	NO	YES	YES
Laboratory Control Std. (LCM,LCT)	NO	YES	YES	YES	YES	NO	YES
Laboratory Reagent Blank (LRB)	NO	YES	YES	YES	NO	NO	NO

SECTION 5

FUNCTIONAL DESIGN

5.1 OVERALL STRUCTURE OF THE AUTOMATED INSTRUMENT SYSTEM

The Standard Calibration Instrument Automation System is a four tiered command level structure as shown in Figures 2, 3a, 3b, and 3c. Level 0 is the SFC Master Command Level (SFCMCL) and its purpose is to facilitate the access of backlog sample data, the development of a Run File or wheel pattern, the automated analysis of samples, and the transmission of sample results to the data basing computer (Figure 2). Level 1 is the Automation System Command Level (ASCL) and its purpose is to assist the analyst in performing pre- and post-run functions as well as initiating an automated run sequence (Figure 3a). Level 2 is the Instrument Run Command Level (IRCL) and it is designed to assist in the measurement of a batch of samples and the reporting of results to the analyst (Figure 3b). Level 3 is the Finish Command Level (FCL) and its purpose is to generate final results for the particular group of samples (Figure 3c). A parallel plotting function is included in the system and its purpose is to facilitate the "off line" plotting of calibration curves to appropriate graphics display terminals (Figure 3d).

5.1.1 SFC Master Command Level (0)

SFC Master Command Level (SFCMCL) will be the first option level encountered by an analyst logging into an instrument system directory. All exits from the system (logging off the computer) will be performed at this level. Figure 2 shows the ten major options available at SFCMCL. Each function will be described briefly.

BACKLOG EDIT OPTION facilitates the access of backlogged samples and the transfer of selected samples to the user's work area. The TEMPLATE EDIT option will aid in the development of skeletal wheel pattern segments describing the frequency and order of analytical and quality control samples. The RUN CREATE option allows the user to combine pre-defined templates and backlog samples into a Run File or Sample Wheel Pattern. The Background Que (BGQUE) option allows an analyst or system manager to que up a background batch job. The MANUAL RUN Option assists an analyst in the process of manually entering sample results into a Run Results File for transfer to the data base computer. The AUTORUN option will facilitate entry to the Automation System Command Level (ASCL). The main function of AUTORUN is to reduce instrument data to final results and use these data to convert a Run File (no calculated results) into a Run Results File (includes calculated results). The RUN EDIT option will assist in the post run modification of Run Results Files including reductions in result

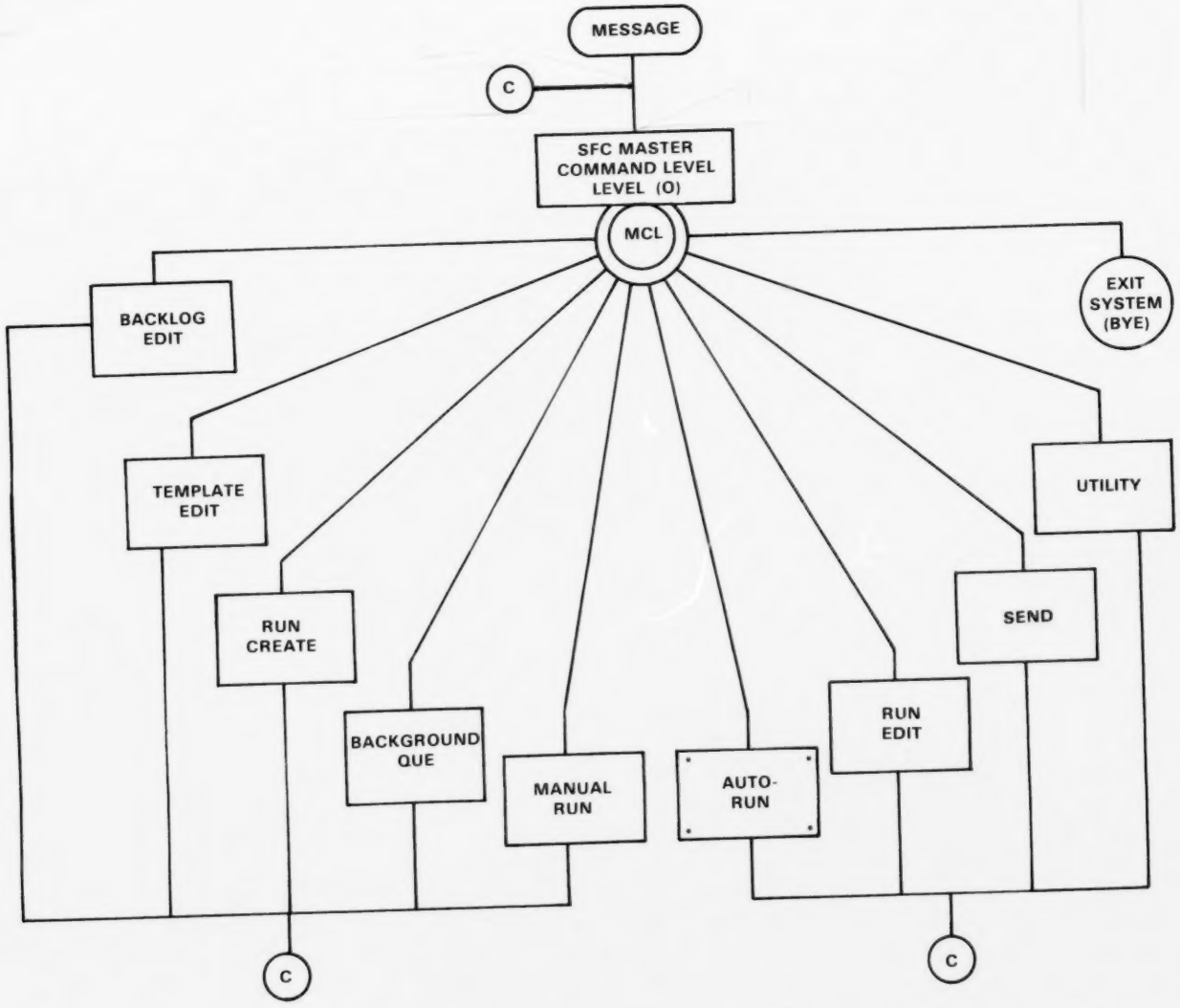
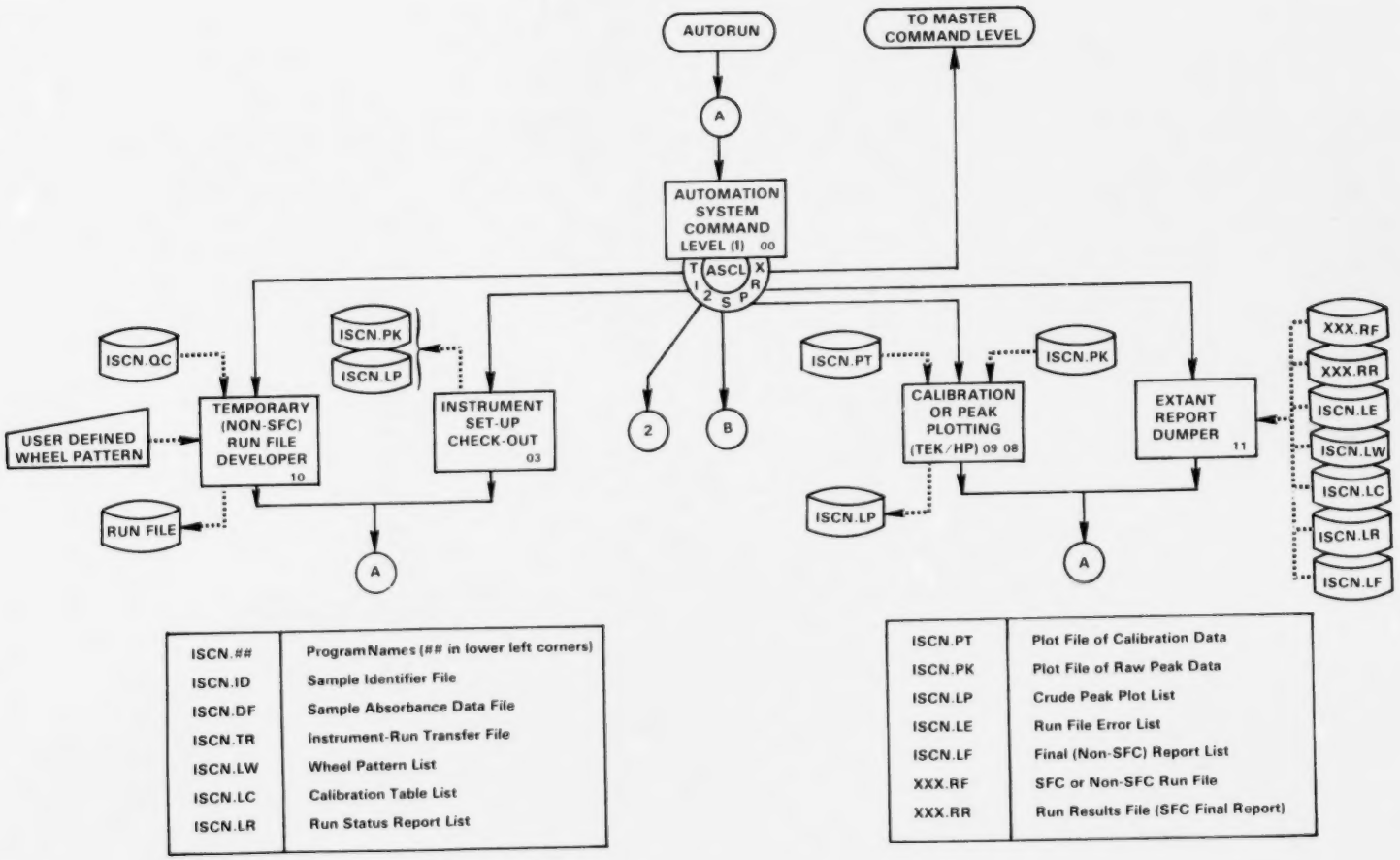


Figure 2. Simplified foreground Basic design for automated instrument system.



ISCN.##	Program Names (## in lower left corners)
ISCN.ID	Sample Identifier File
ISCN.DF	Sample Absorbance Data File
ISCN.TR	Instrument-Run Transfer File
ISCN.LW	Wheel Pattern List
ISCN.LC	Calibration Table List
ISCN.LR	Run Status Report List

ISCN.PT	Plot File of Calibration Data
ISCN.PK	Plot File of Raw Peak Data
ISCN.LP	Crude Peak Plot List
ISCN.LE	Run File Error List
ISCN.LF	Final (Non-SFC) Report List
XXX.RF	SFC or Non-SFC Run File
XXX.RR	Run Results File (SFC Final Report)

Figure 3a. Simplified RUN automated instrument system (ASCL).

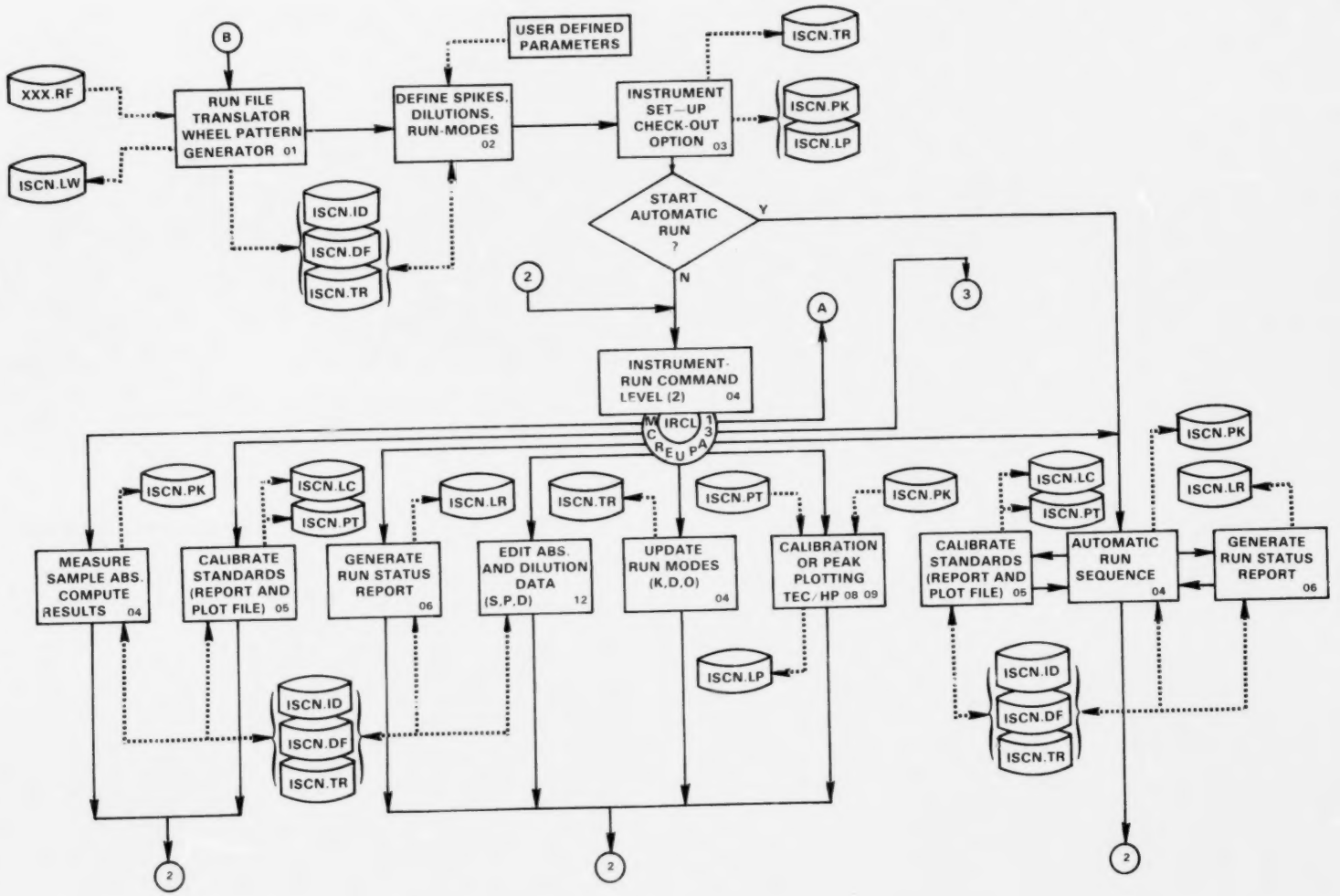


Figure 3b. Simplified RUN automated instrument system (IRCL).

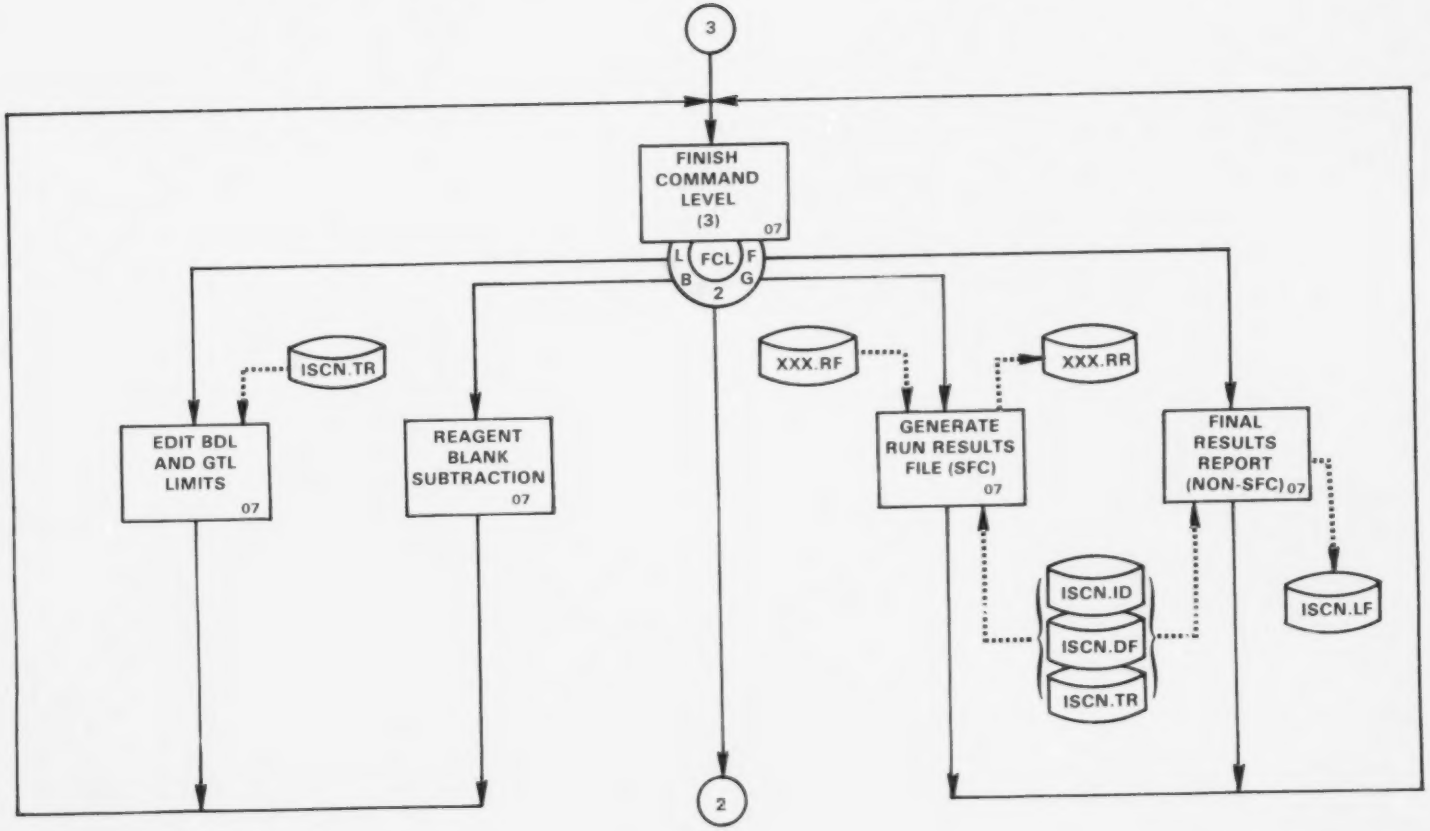


Figure 3c. Simplified RUN automated instrument system (FCL).

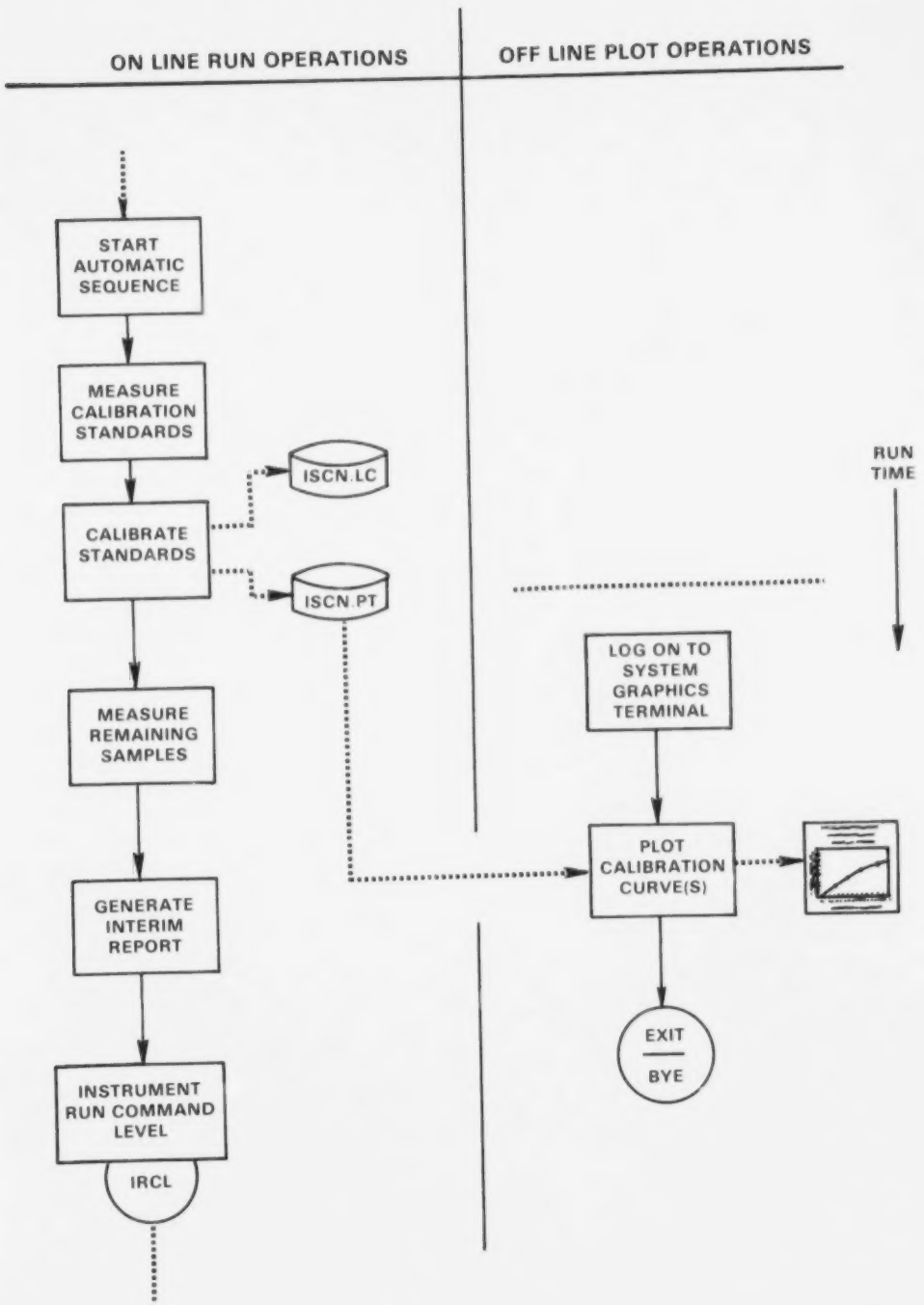


Figure 3d. Off-line calibration plotting capabilities.

significance as well as the attachment of non-numeric qualifiers (BDL = below detection limit) to specified samples. The SEND option will utilize PRJE (PDP 11/70 Remote Job Entry) to que a batch job to the PDP 11-70 which contains a Run Results File from a manual or automatic run. The UTILITY option will offer the analyst a menu of approved programs to select from. These programs may include statistical programs or other progrms needed by the analyst. The EXIT option terminates the session and automatically BYEs out of the system. The EXIT option will automatically be executed if a certain time limit has expired without a user response.

Further documentation will soon be available on the design and operation of the SFCMCL options.[8] The remainder of this document will be dedicated to describing the design of the SFCMCL AUTORUN option.

5.1.2 Automation System Command Level (1)

Automation System Command Level is accessed by SFCMCL and can exit to SFCMCL on command or through a timed exit. Figure 3a depicts the ASCL options and illustrates the files utilized in the automation system.

The majority of ASCL options are pre- and post-run utilities. The Temporary (Non-SFC) Run File Developer is available for creation of a Run File. It has been designed to serve in the place of TEMPLATE EDIT and RUN CREATE during the development phase of the SFCMCL system. The Temporary Run File Developer will be useful for expediting the creation of a Run File in purely test or research situations. This module allows for the isolation of the Automated Instrument System from the SFCMCL system.

One important function of the Temporary Run File Developer is that it facilitates the maintenance of quality control limits required by the automated instrument system. As QC samples are defined for a run, the appropriate limits are accessed and supplied to the Run File as limit records. The format of these records will be identical to those supplied by the RUN CREATE option of the SFCMCL. An analyst will be given the opportunity to update these limits prior to the development of a Run File. Only one set of limits will be maintained for each quality control type (Lab Reagent Blank, Lab Control Standard, Lab Duplicate or Lab Spike).

The Instrument set up/check out module may be accessed external to a run situation for the evaluation of the interfaced instrument. The module is also available as a sequential part of the automated instrument run as described in Section 5.2.2 and depicted in Figure 3b.

The Calibration Curve and peak Array Plotting module will facilitate the display of curves and raw data arrays accumulated during the last automated instrument run. If the instrument requires raw peak data acquisition and reduction, then the peak data will be retrievable for every sample injection made in the run. Data will be accessed, sequentially. If the instrument supplies a fully reduced absorbance as the result of an injection, then no raw peak data will be available. Calibration curves will be available irrespective of what type of instrument is used. Since calibration will be

performed on a multi-curve basis, as many as five curves may be previewed in a session. The output device must be either a Tektronix or a Tektronix compatible graphics terminal for calibration curve plotting, however, peak plotting will be available in crude form on a hardcopy terminal or the lineprinter if a graphics terminal is not available.

The Extant Report Dumper is another post-run utility which can regenerate any of the numerous sequential list files generated during a run:

- wheel pattern list (ISCN.LW)
- calibration report (ISCN.LC)
- interim report (ISCN.LR)
- final report (ISCN.LF)
- run file translator error list (ISCN.ID)
- sample absorbance data file (ISCN.DF)
- instrument run transfer file (ISCN.TR)

It may even be used to output Run Files, Run Results Files, or any 80 byte sequential list file to the terminal or lineprinter.

Two options are available for entering Instrument Run Command Level (IRCL). The normal entry will require pre-run processing through three modules shown in Figure 3b and described in Section 5.2.3. Another entry to IRCL (level 2) is available for analysts who wish to resume a previous run and maintain current run data. For example, if an analyst completed an automated run and exited the automation system, he could reenter the system at a later time and perform the post-run processing necessary to produce an acceptable Run Results File or final report. Under certain circumstances, this direct access route will be unavailable as in the case where SFC files have been modified, deleted or sent to the data basing computer during the intervening time.

5.1.3 Instrument Run Command Level (2)

The bulk of the automated instrument system capabilities are accessible through IRCL. These functions are depicted in Figure 3b and are described in more functional terms in Section 5.2. Essentially the system is designed as a discrete measure/edit single-analyte standard calibration system which may be used to perform an analysis run on a sample-by-sample basis or perform an automatic sequence of sample measurements on command.

5.1.4 Finish Command Level (3)

Section 5.3 of this document will describe the functional aspects of the FCL (Figure 3c). The major purpose of the FCL is to produce a Run Results File for SFC related runs or a "clean" final report for non-SFC applications. This module is accessed from IRCL and returns to IRCL. It is designed to be reentrant within the context of the same run. That is, a Run File may be converted to a Run Results File without closing off the option of performing additional measure/edit functions and reformulating the Run Results File.

5.2 INSTRUMENT RUN COMMAND LEVEL FUNCTIONS

5.2.1 Pre-Run Method Definition

Pre run method definition will be the first phase in any automated analysis (see Figure 3b). Within this phase, a number of tasks will be performed. The first of these is the system verification of the Run File. The analyst will be required to enter the name of the Run File which identifies the sample sequence he desires to run. The system will then verify the run sequence. As the system verifies the Run File, it will create and store run data in an instrument compatible data file (ISCN.DF) and a sample identifier file (ISCN.ID). ISCN.DF will be used throughout the run to store raw absorbance data on samples as they are measured. If the Run File produces errors due to form or content, the file will be output with the erroneous record identified and the program will exit to Automation System Command Level (Figure 3a). If the Run File is acceptable, a sample wheel pattern will be output to the terminal or lineprinter.

A transfer file (ISCN.TR) will serve as a main data file for operator inputs, instrument parameters, and calibration data acquired over the course of a run. During run verification the number of standards, unknowns and QC items will also be stored in the transfer file. The transfer file, the ID file, and the raw data file will be in the Read/Write or random access format.

Within the pre run method definition phase, the analyst will be required to answer a number of questions regarding the initial run conditions. Information that remains the same from one run to the next will not need to be input each time. Some types of preliminary data include the analyst's initials, analyte being measured, units of concentration, computational mode, dilution factors, spiked sample volumes, and general pre run comments.

In addition to these items, the user will be required to specify whether the run is to start in the fully automatic or semi automatic mode. If the run is fully automatic, the system will request the number of replicate injections required for each sample.

The default output mode must also be specified prior to initiating the run. Since calibration and status reports will be generated automatically in a fully automated sequence, the system must know the output mode, that is, terminal, lineprinter, both or neither.

One of the most important pre run modes is the data acquisition mode. In most instrument automation systems, data is automatically acquired and reduced by the computer. In some cases, however, an analyst may choose to enter sample absorbances manually through the keyboard. This data may be read off strip chart recorders or digital display meters and the analyst will type the numbers into the terminal in order to utilize the computational and reporting power of the standard calibration system.

5.2.2 Instrument Initialization and Check-out

An optional instrument initialization/check-out procedure may be performed by the analyst immediately before an automated run. It is assumed, here, that the instrument has been turned on and the source lamps have been stabilized before this phase begins. The system will cycle through a check list to assist the analyst in setting the initial operating conditions, and will then automatically check the connection between the computer and the instrument.

In instrument systems which require raw peak data acquisition and reduction, the system will prompt the user to input the instrument atomization time and the analyst may make a test injection to verify the data acquisition timing. A crude plot may be output at the line printer or terminal showing the test peak and the analyst may change the timing parameters. In systems where the instrument itself produces a fully reduced absorbance value, the analyst will be given the opportunity to make test injections in order to verify interface reception and transmission of the output character string.

5.2.3 Sample Measurement

The sample measurement function is the most basic element of the automated run. A wheel location is the only information needed by the system to perform the measurement since the system maintains a Wheel Pattern showing what samples are in each location. The system will first output the sample descriptor. The system will then collect data from the atomization. This data will be displayed at the user terminal as an absorbance value. Additional computations will be made and displayed if the item being measured is an unknown sample or a quality control sample. Once the sample information has been displayed, the system will be ready to accept the next measurement instruction. In a fully automatic run, these instructions will be automatically generated by the system software until all of the samples in the sample wheel have been measured. In a semi-automatic run, the instructions must be manually entered by the analyst.

5.2.4 Calibration and Computation

The analyst will be responsible for making a preliminary decision as to the type of equation which will best fit the standard points measured in a standard calibration analysis. After the last standard is measured during a fully automatic sequence, the system will automatically perform the calibration of standard's data. The result of this calibration will be a set of equations representing the five possible computational modes -- forced-zero linear, regular linear, quadratic, cubic, and interpolation. The following equations represent the relationship between concentration and absorbance as obtained through least squares regression:

Forced Zero: $conc = a_0 \times ABS,$

Regular Linear: $conc = a_1 + a_2 \times ABS,$

Quadratic: $conc = a_3 + a_4 \times ABS + a_5 \times ABS^2,$

Cubic: $conc = a_6 + a_7 \times ABS + a_8 \times ABS^2 + a_9 \times ABS^3.$

In cases where excessive curvature occurs, the equations may produce erroneous results and the analyst may elect to compute concentrations through the process of linear interpolation between two bracketing standards. The system will be unable to produce a fitting error when interpolation is used to compute sample concentrations.

In the process of calibrating the standards, this system will develop equations for all optional computational modes, with the only constraint being the number of standards measured. The system will display the equation and the fitting error associated with the chosen calibration mode. The fitting error or percent relative standard error of estimate will be computed and displayed. A table will be produced showing the known concentration, the average measured absorbance, the computed concentration using the appropriate equation, and the difference between known and calculated concentrations. This table and the associated fitting error will appraise the analyst of discrepancies encountered in calibration. Figure 4 shows an example of the standard calibration report. In addition, plots of standard points and equations may be produced to fully depict the standards (see Figure 5 and Section 5.2.10 below).

During the calibration process, the system will record the absorbance and concentration for the highest and lowest non-zero calibration standards used in the calibration. This information will be used in subsequent sample determinations in order to alert the analyst to any off scale sample measurements.

The chosen mode of computation will be used throughout a fully automatic run to compute unknown and quality control sample concentrations. The analyst may change the computational mode at any time during semi automatic sequencing. In addition, the analyst may perform a complete recalibration of standards from the Instrument Run Command Level if the standard's data has been modified by editing or remeasurement.

5.2.5 Interim Reporting

The instrument report will automatically be generated at the end of a fully automatic run and will contain all of the essential information on each sample measured during the run. An instrument report will indicate which samples were off scale. It will also indicate problems encountered in standard calibration and will signal all limit violations for quality control samples. After a report is generated, control will be returned to the IRCL. The analyst may generate an interim report at any time from IRCL. An example of a standard calibration interim report is shown in Figure 6.

5.2.6 Fully Automatic Analysis Sequence

A fully automatic sequence may be performed on a set of samples if an automatic sampling device has been installed on an instrument . The only operator intervention needed during an automatic sequence will be the changing of a sample wheel when all of the samples in the first wheel have been measured. Normally, Perkin-Elmer sample changers (model AS-1) have

CALIBRATION STANDARD'S TABLE

MODE: QUADRATIC
NUMBER OF STDS: 7
FITTING ERROR: 4.80 %

TIME: 15:33 ON 5/19/1980
HIGHEST STD: 100
LOWEST STD: 5

EQUATION: CONC=-2.19E-02+3.47E+02*ABS+9.25E+00*ABS^2

WHEEL POS		STANDARD IDENTIFIER	NO. REPS	AVE ABS	CONC EXPECT	CONC FOUND	DIFF
1	3	+0.000E+00	1	+0.0010	+0.00	+0.33	-0.33
2	3	+5.000E+00	1	+0.0190	+5.00	+6.57	-1.57
3	3	+1.000E+01	1	+0.0240	+10.00	+8.31	+1.69
4	3	+2.000E+01	1	+0.0540	+20.00	+18.74	+1.26
5	3	+3.000E+01	1	+0.0900	+30.00	+31.28	-1.28
6	3	+5.000E+01	1	+0.1430	+50.00	+49.79	+0.21
7	3	+1.000E+02	1	+0.2860	+100.00	+99.98	+0.02

Figure 4. Flameless atomic absorption calibration report.

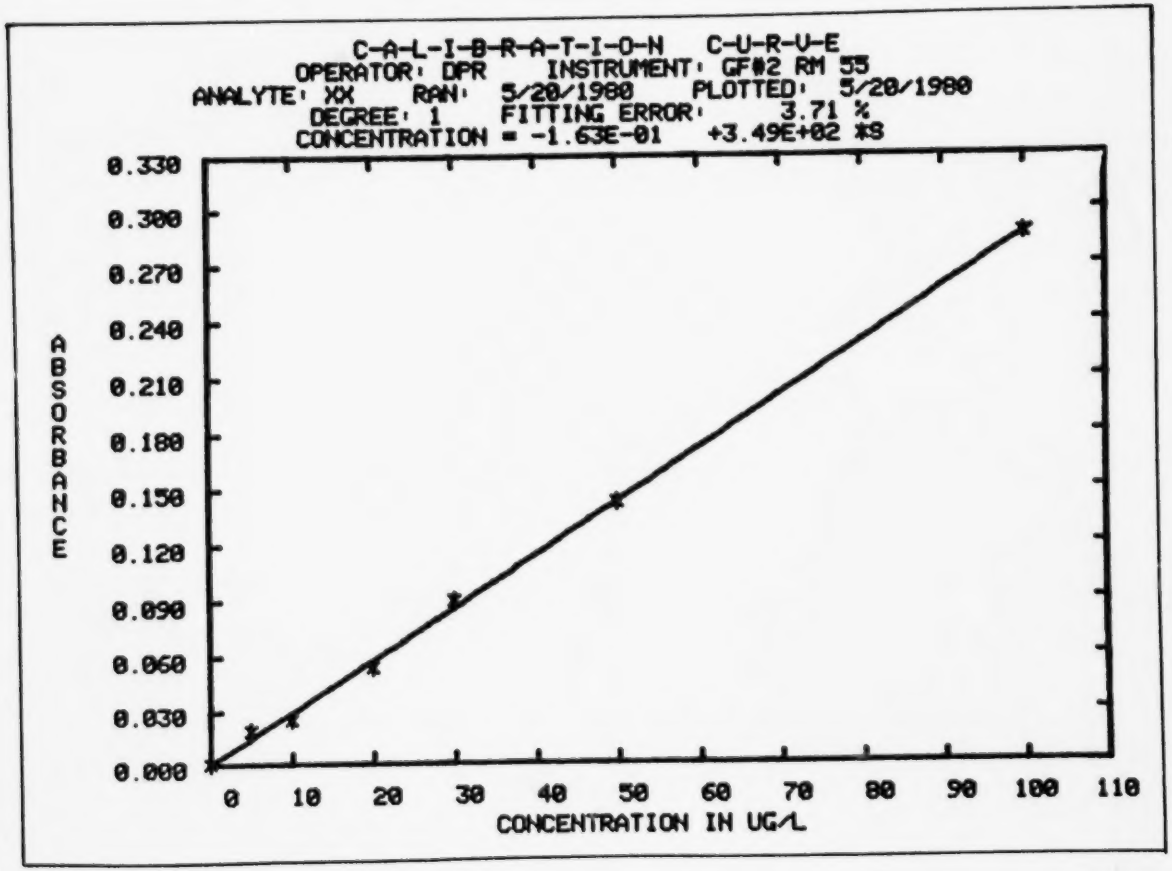
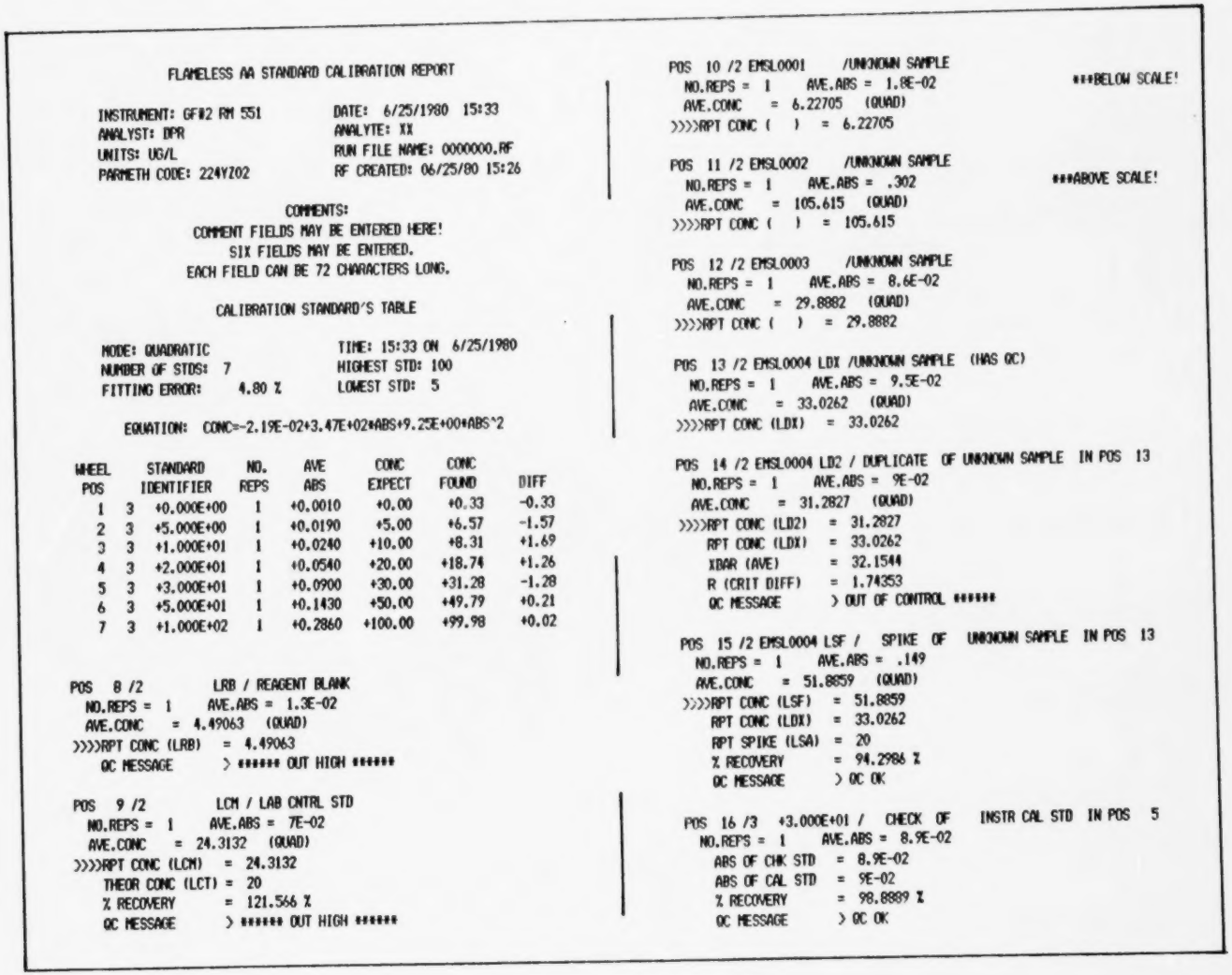


Figure 5. Flameless atomic absorption calibration curve plot.



```
                FLAMELESS AA STANDARD CALIBRATION REPORT

 INSTRUMENT: GF#2 RM 551          DATE:  6/25/1980  15:33
 ANALYST: DPR                     ANALYTE: XX
 UNITS: UG/L                      RUN FILE NAME: 0000000.RF
 PARMETH CODE: 224YZ02            RF CREATED: 06/25/80 15:26

                        COMMENTS:
                COMMENT FIELDS MAY BE ENTERED HERE!
                   SIX FIELDS MAY BE ENTERED.
                EACH FIELD CAN BE 72 CHARACTERS LONG.

                    CALIBRATION STANDARD'S TABLE

 MODE: QUADRATIC                  TIME: 15:33 ON  6/25/1980
 NUMBER OF STDS:  7               HIGHEST STD: 100
 FITTING ERROR:     4.80 %        LOWEST STD:  5

    EQUATION:  CONC=-2.19E-02+3.47E+02*ABS+9.25E+00*ABS^2

WHEEL    STANDARD      NO.    AVE       CONC      CONC
 POS    IDENTIFIER     REPS   ABS      EXPECT    FOUND      DIFF
  1   3  +0.000E+00     1   +0.0010     +0.00     +0.33     -0.33
  2   3  +5.000E+00     1   +0.0190     +5.00     +6.57     -1.57
  3   3  +1.000E+01     1   +0.0240    +10.00     +8.31     +1.69
  4   3  +2.000E+01     1   +0.0540    +20.00    +18.74     +1.26
  5   3  +3.000E+01     1   +0.0900    +30.00    +31.28     -1.28
  6   3  +5.000E+01     1   +0.1430    +50.00    +49.79     +0.21
  7   3  +1.000E+02     1   +0.2860   +100.00    +99.98     +0.02


POS   8 /2          LRB / REAGENT BLANK
  NO.REPS =  1     AVE.ABS =  1.3E-02
  AVE.CONC    =  4.49063   (QUAD)
>>>>RPT CONC (LRB)    =  4.49063
    QC MESSAGE        > ****** OUT HIGH ******

POS   9 /2          LCM / LAB CNTRL STD
  NO.REPS =  1     AVE.ABS =  7E-02
  AVE.CONC    =  24.3132   (QUAD)
>>>>RPT CONC (LCM)    =  24.3132
    THEOR CONC (LCT) =  20
    % RECOVERY       =  121.566 %
    QC MESSAGE        > ****** OUT HIGH ******

POS  10 /2 EMSL0001      /UNKNOWN SAMPLE
  NO.REPS =  1     AVE.ABS =  1.8E-02                    ***BELOW SCALE!
  AVE.CONC    =  6.22705   (QUAD)
>>>>RPT CONC (   )    =  6.22705

POS  11 /2 EMSL0002      /UNKNOWN SAMPLE
  NO.REPS =  1     AVE.ABS =  .302                       ***ABOVE SCALE!
  AVE.CONC    =  105.615   (QUAD)
>>>>RPT CONC (   )    =  105.615

POS  12 /2 EMSL0003      /UNKNOWN SAMPLE
  NO.REPS =  1     AVE.ABS =  8.6E-02
  AVE.CONC    =  29.8882   (QUAD)
>>>>RPT CONC (   )    =  29.8882

POS  13 /2 EMSL0004 LDI /UNKNOWN SAMPLE  (HAS QC)
  NO.REPS =  1     AVE.ABS =  9.5E-02
  AVE.CONC    =  33.0262   (QUAD)
>>>>RPT CONC (LDI)    =  33.0262

POS  14 /2 EMSL0004 LD2 / DUPLICATE  OF UNKNOWN SAMPLE  IN POS  13
  NO.REPS =  1     AVE.ABS =  9E-02
  AVE.CONC    =  31.2827   (QUAD)
>>>>RPT CONC (LD2)    =  31.2827
    RPT CONC (LDI)    =  33.0262
    XBAR (AVE)        =  32.1544
    R (CRIT DIFF)     =  1.74353
    QC MESSAGE        > OUT OF CONTROL ******

POS  15 /2 EMSL0004 LSF /  SPIKE  OF  UNKNOWN SAMPLE  IN POS  13
  NO.REPS =  1     AVE.ABS =  .149
  AVE.CONC    =  51.8859   (QUAD)
>>>>RPT CONC (LSF)    =  51.8859
    RPT CONC (LDI)    =  33.0262
    RPT SPIKE (LSA)   =  20
    % RECOVERY        =  94.2986 %
    QC MESSAGE        > QC OK

POS  16 /3  +3.000E+01 /  CHECK  OF   INSTR CAL STD  IN POS   5
  NO.REPS =  1     AVE.ABS =  8.9E-02
    ABS OF CHK STD   =  8.9E-02
    ABS OF CAL STD   =  9E-02
    % RECOVERY       =  98.8889 %
    QC MESSAGE        > QC OK
```

Figure 6. Typical interim report.

thirty positions. If a sample wheel pattern has more than thirty positions defined for the run, the system will measure the first tray of samples and wait for the second tray to be loaded by the analyst. When the analyst has performed the task, he will restart the changer and the run will continue until the last sample defined in the sample wheel pattern is measured.

During the fully automated run the system will perform calibration after the standards have been measured, will generate measurement information after each sample atomization, and will generate an interim report after the last sample has been measured. It will also allow for up to six replicate measurements of the same sample before cycling to the next sample. After a report has been generated the system will revert to the semi automatic operator controlled mode at IRCL. Fully automatic sequencing may easily be resumed at the chosen wheel position by manually repositioning the wheel, resetting the replicate counter, and using the command for restarting an automatic sequence.

5.2.7 Quality Control Assessment

The proposed Sample File Control system will supply quality control limits for every distinct method parameter code in a sample backlog listing. As an analyst defines QC items for his Run File, the SFC system will select the appropriate limits and assign the values to the sample in the form of limit records. These limits will be stored in the instrument data file during Run File translation, and will be referenced during a run to assess QC acceptability. The system will sound an alarm if warning or control limits are exceeded during a run. If the analyst wishes to suspend a fully automatic run because of control violations, he may do so by pressing the escape key and the system will revert to the operator controlled semi automatic mode of operation at IRCL and await instructions.

For systems not utilizing the SFCMCL QC limits, provisions will be made to allow hand entry of QC limits during the Run File Development process located at the ASCL. (See 5.1.2)

5.2.8 Sample Editing

Sample editing will be available after an automatic run has been completed and the first interim report has been generated. Three different edit functions have been designed for the system and they include the ability to selectively delete a replicate absorbance for a specified sample, the ability to purge all absorbance data for one or more contiguous samples in the wheel pattern, and the ability to modify dilution volumes for samples requiring dilution and remeasurement.

The selective replicate delete capability will allow for the preview of all information available for any sample in the wheel pattern. The analyst may delete replicate absorbance data before returning to IRCL. A special message will be generated if standard's data is modified indicating the need for a recalibration at the next available opportunity.

The purge capability facilitates the erasure of all replicate data stored for specified groups of samples. If a problem occurs in an automatic sequence, the analyst will be able to stop sampling, repair the problem, purge erroneous data on the affected samples, reposition the wheel and restart an automatic sequence at the appropriate position.

If a sample measures off scale with respect to the highest calibration standard, the analyst will be given the opportunity enter the appropriate dilution volumes, dilute, and remeasure the sample.

5.2.9 Run-Mode Options

The ability to modify Run-Modes will be available to the analyst at IRCL. The three Run-Modes are the computational mode, the output mode, and the data acquisition mode.

The five computational modes are forced-zero linear, regular linear, quadratic, cubic, and interpolation. The capability of changing the computational mode will involve only a flag change and not a recalibration. For example, if the analyst chooses the regular linear computational mode prior to an automatic sequence, then all sample concentrations will be computed with the linear equation relating standard concentration to the measured absorbance. During the run and after standards have been calibrated, the analyst may review the multiple calibration curves on the system graphics terminal (5.2.10) and decide that the quadratic mode is more suitable for the analysis. The interim report will be generated using the previously defined regular linear mode but when IRCL control is returned to the analyst, he may quickly change the mode from linear to quadriatic without recalibrating and then generate another interim report with revised determinations. All terminal output for a sample will be based on the current computational mode and will be qualified with a computational mode identifier.

The analyst will have the opportunity to change the output mode from the IRCL. The options include outputting interim reports, calibration reports, and final reports to the lineprinter, the terminal, both lineprinter and terminal, or neither lineprinter or terminal. The actual on-line terminal output will not be affected by the change in output mode. The ability to route output to the chosen device will save time and assist in the conservation of system resources.

A very special feature has been designed to give an analyst the capability to change the data acquisition mode at the IRCL. This option will allow the analyst to switch from an automatic data acquisition and reduction mode to a manual or hand entry mode and vice versa. An example of the correct usage of this feature would be when an analyst has made a mistake filling his sample wheel and the sample measurements are out of sequence. The analyst who detects this problem may stop the automatic run, generate an interim report, purge the data for the affected samples, change the data acquisition mode to hand entry, and transcribe the results from the

report to the proper samples. The mode may then be changed back to automatic data acquisition and the run may be restarted from a selected position. The ability to switch from hand entry to automatic data acquisition mode will not always be available especially when the run was started in the hand entry mode. Automatic sequencing will still be available irrespective of what data acquisition mode is in use.

5.2.10 Plotting Capabilities

The system has been designed to facilitate plotting of calibration curves and raw data peaks. This feature is redundant in order to allow for maximum usage of the capability irrespective of the location and type of graphics display devices available to the analyst.

A primary assumption regarding plotting is that a laboratory automation system will have at least one Tektronix or Tektronix compatible graphics terminal available to all users. In addition, some analysts may share a graphics terminal with several users or have a dedicated instrument terminal with appropriate graphics capabilities. In this last case, an analyst will be able to display calibration curves and peak plots within the context of the Instrument Run Command Level (IRCL).

The major plotting capability centers around the display of the calibration curves (Figure 5). A special feature of the system allows an analyst to plot his calibration curves during an automated run immediately after calibration has been performed. Figure 3d depicts the parallel nature of the process. Each analyst will be given a plot account identifier which may be used to generate calibration curves on the shared graphics terminal. By reviewing these curves, the analyst can easily decide if the chosen computational mode was correct or if a computational mode change is required. This special case application will be available for all systems which perform calibration on standards.

Raw peak data plotting will be available in those systems where the instrument produces an analog voltage signal during atomization. Figure 7 shows an example of a peak plot. Each partially reduced atomization array will be stored during the course of a run and may be plotted (after the run is completed) on the shared graphics device through the Automation System Command Level plot-option (see Section 5.1.3). In general, this is a research tool and will be very useful in analyzing factors affecting atomization. Erratic instrument behavior can be visually located and conditions can be corrected. Peak plotting is usually performed external to a run but for those with dedicated graphics devices, peaks as well as calibration curves may be reviewed through the IRCL plot option.

5.3 Finish Command Level Options

Finish Command Level (3) is accessible from Instrument Run Command Level (2) and exists to the IRCL. There are two major functions of the FCL and they are to facilitate the formation of a Run Results file for SFC related analyses and to prepare a "clean" final report for non-SFC analyses. The generation of either type of report will be possible irrespective of the

FLAMELESS AA PEAK ARRAY PARAMETERS (GSC1)

DATE: 4/ 3/1980 - 16:26:38	MEASURED: 3/ 7/1980 - 9: 3:29
ANALYST: TLR	ANALYTE: PB
SAMPLE TYPE: TIMING SET STD	SAMPLE ID:
PEAK NO: 1	WHL POS: 0
INSTRUMENT:	MODE: FULLY-AUTO STD CAL
METHOD: PEAK HEIGHT	BASELINE: LEADING
A/D CHANNEL: 0	NUMB PTS IN CALL: 300
DELAY BEFORE: 857*(1/60)	TIME BETWEEN PTS: 2*(1/60)
NUMB ACTUAL PTS: 300	
MAX ARRAY VALUE: -0.6340	MAX LOCATION: 156
MIN ARRAY VALUE: -0.7384	MIN LOCATION: 3
AVE LEADING BSLN: -0.7378	BSLN STD DEV: 6.529E-04
PEAK HEIGHT: 0.1039	PEAK LOCATION: 156.20

PLOT PEAK (N[CR] OR Y)) Y

F-U-R-N-A-C-E P-E-A-K D-A-T-A P-L-O-T

PLOTTED: 4/ 3/1980 - 16:28:39 RAN: 1/ 0/ 0 - 9: 3

PEAK NO: 1 TYPE: TIMING SET STD WHL POS: 0

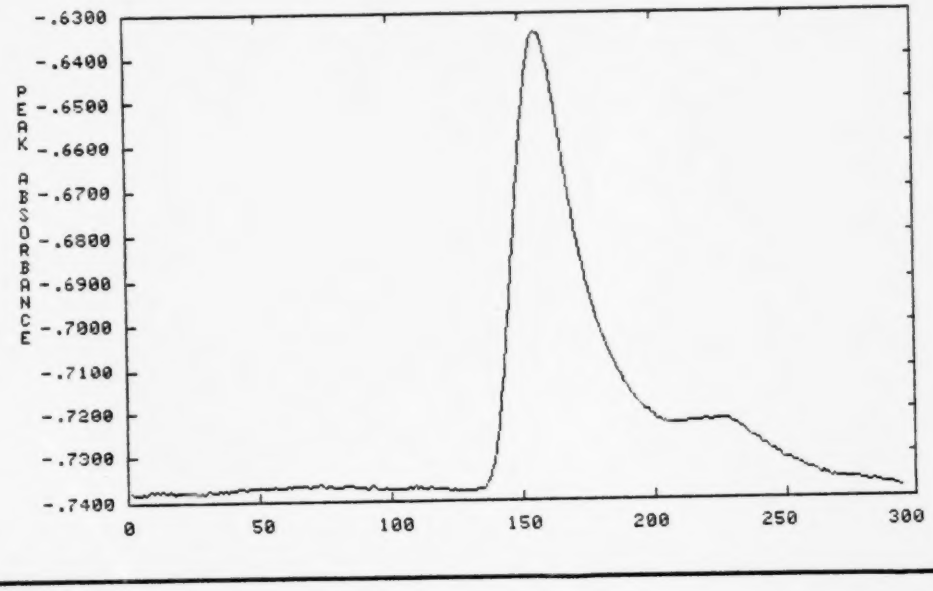


Figure 7. Furnace peak data plot.

sample file control aspects of the run. Both reports will contain sample identifiers, sample concentrations, and non-numeric qualifiers. (All detailed information on the sample will be available in the interim report.) See Figure 8 for an example of a final output report.

In addition to the generation of the two types of final reports, the system will be capable of aiding the analyst in performing two important pre-report functions, these are the editing of BDL/GTL limits and the adjustment of sample levels through reagent blank subtraction.

BDL is a sample qualifier which is assigned to those environmental samples which have computed concentrations below a specified determination level. GTL is a sample qualifier which is assigned to those environmental samples which have computed concentrations in excess of a specified maximum determination level. The report generating mechanism of the FCL will assume that all samples with computed concentrations which fall below the lowest non-zero calibration standard concentration level will be flagged with a BDL and that all samples with computed concentrations which exceed the highest calibration standard concentration level will be flagged with a GTL. By using the Finish Command Level BDL/GTL edit capability, the analyst can modify the range of these limits for subsequent report generation. During an analysis run, all samples which measure higher than the highest standard will be brought to the attention of the analyst. The analyst is solely responsible for deciding whether a sample needs additional dilution and remeasurement or can be accepted without qualification as determined.

Another feature of the FCL is that it will facilitate the formation of a reagent-blank signal subtraction array. Because of the heuristic nature of the means by which reagent contamination is determined, a highly flexible technique has been devised for performing reagent subtraction and fulfilling SFC requirements. This is done by giving the analyst complete control in choosing groups of samples and assigning reagent signal levels to each group as deemed necessary. No distortion of real absorbance data will occur and in the subsequent report generation stage, the assigned signal levels will be subtracted from the applicable sample before the sample concentration is computed.

FLAMELESS AA STANDARD CALIBRATION FINAL REPORT

INSTRUMENT: GF#2 RM 551
ANALYST: DPR
UNITS: UG/L
PARMETH CODE: 224YZ02

DATE: 5/19/1980 15:39
ANALYTE: XX
RUN FILE NAME: 0000000.RF
RF CREATED: 05/19/80 15:38

WHEEL POS	SAMPLE IDENTIFIER	SAMPLE CONCENTRATION	KNOWN SPIKE OR CONTROL	VIOLATION LIMIT	QC
8	LRB	+4.4906			OUTC
9	LCM	+24.313	+20.00		OUTC
10	EMSL0001	+5.5093			
11	EMSL0002	+102.09			
12	EMSL0003	+26.403			
13	EMSL0004 LDX	+29.539			
14	EMSL0004 LD2	+27.797			OUTC
15	EMSL0004 LSF	+48.389	+20.00		OK

Figure 8. Typical final output report.

SECTION 6

SIGNOFF SHEET

These documents, which describe a proposed automated flameless atomic absorption instrument system but do not constitute an implementation design, are approved by the undersigned interested parties with the understanding that changes in detail are likely and that implementation of all features depends upon the availability of funding and personpower.

Requestors:	_____	Ted Martin, EMSL, Cinti.
	_____	John Morris, Region V, S&A
	_____	Pat Johnson, Region III, S&A
	_____	Tom Reed, MERL, Cinti.
	_____	James Caldwell, MERL, Cinti.
Implementors:	_____	Dennis Ryan and John Teuschler
Flameless Atomic Absorption Instrument System Project Officer:	_____	John Teuschler
Laboratory Automation Project Officer:	_____	William Budde

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